2.2.1 Amination of 1,1-diiodo-2,2-dinitroethene 33

2.2.2 Reaction of Hetero-cyclic Compound 34

2.2.3 Reaction of Methylimidazole 35

2.2.4 Reaction of Methoxy-methylimidazoledine-dione 36

2.2.5 Reaction of Methyl-Pyrimidine Dione 37

2.3 Selection of FOX-7 Synthesis Route 39

2.4 Reaction Mechanism 40

2.5 Nitration Process Optimization 41

2.5.1 Experimental & Discussion 42

2.5.1.1 Effect of moles of sulphuric acid 43

2.5.1.2 Effect of moles of nitric acids 44

2.5.1.3 Effect of temperature on solubility MDP 45

2.5.1.4 Nitration temperature domain 46

2.5.1.5 Effect of temperature on the yield of FOX-7 47

2.5.1.6 Effect of concentration on nitric acid 49

2.6 Hydrolysis process optimization 49

2.6.1 Optimization of acid (%) for hydrolysis 51

2.6.2 Optimization of Hydrolysis temperature & time 51

2.7 Conclusions 53
3. Thermo-chemical studies of MDP nitration process

3.1 Introduction

3.1.1 Calorimetry in thermal hazard quantification

3.1.2 Heat flow/reaction calorimetry

3.1.3 Power compensation/reaction calorimetry

3.2 Thermal Hazards in MDP Nitration

3.3 Experimental

3.3.1 Chemicals & Samples

3.3.2 Reaction

3.4 Methodology

3.4.1 Differential scanning Calorimetry

3.4.2 Experiments in Reaction Calorimetry

3.4.2.1 Nitration of MDP Experiments in RC (Mettler)

3.4.2.2 Nitration of MDP Experiments in RC (HEL)

3.5 Results and Discussion

3.6 Conclusion

3.7 References
4. Reaction Kinetics & Parametric sensitivity for FOX-7 synthesis process

4.1 Introduction

4.2 Nitration kinetics

4.2.1 Nitration Reaction

4.2.2 Nitration Experiments

4.2.3 Analysis of Kinetic data

4.2.3.1 Reaction Rate

4.2.4 Temperature dependency rate equation

4.2.5 Estimation of heat generation during Nitration

4.2.6 Results and discussion: Nitration Kinetics

4.2.7 Conclusion: Nitration kinetics

4.3 Hydrolysis kinetics

4.3.1 Hydrolysis Reaction

4.3.2 Hydrolysis Experiments

4.3.3 Hydrolysis Kinetics

4.3.4 Results and discussion: Hydrolysis kinetics

4.3.5 Conclusion: Hydrolysis kinetics

4.4 Studies on Parametric Sensitivity of MDP Nitration

4.4.1 Introduction
4.4.2 Modelling

4.4.3 Experiment

4.4.4 Simulation Results and Analysis of Parametric Sensitivity

4.4.5 Conclusion

4.5 References

5. Micro Reactor Modelling and Simulation with MDP Nitration

5.1 Importance of Micro reactor

5.2 Introduction

5.3 Modeling

5.3.1 Reaction

5.3.2 Micro reactor

5.3.3 Modelling of micro-tubular reactor

5.3.4 Dimensionless Variables

5.3.5 Solution of PDE

5.3.6 Simulation

5.3.7 Simulation results

5.4 Model Validation

5.4.1 Experimental

5.4.1.1 Experiments in micro tubular reactor
5.4.2 Comparison: micro reactor vs batch reactor

5.5 Results and discussion

5.6 Conclusion

5.7 References

6. Studies on Preparation of Spherical Particle of FOX-7 using micellar Nano reactor

6.1 Abstract

6.2 Introduction

6.2.1 Design of Nano reactor

6.3 Experimental

6.3.1 Chemicals

6.3.2 FOX-7 by normal synthesis method

6.3.3 Spherical FOX-7 particle using Micro emulsion method

6.4 Results and discussion

6.5 Conclusions

6.6 References

7. Investigation of Polymorphic Behaviour of FOX-7 Crystal

7.1 Abstract

7.2 Introduction

7.3 Experimental
7.4 Results and Discussion

7.4.1 Single crystal X-ray Diffraction of FOX-7

7.4.2 Hot-stage Microscopy studies

7.4.3 Thermal phase transformation studies

7.4.4 Crystal structure Analysis of FOX-7

7.4.5 Molecular organization upon phase transformation

7.4.6 X-Ray study of \( \gamma \) form of FOX-7

7.4.7 Thermal Studies

7.4 Conclusion

7.5 References

8. Summary

9. Publications